Young's Modulus of Vinyl Ester Composites Cured by Microwave Irradiation: Preliminary Results

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Abstract: The shrinkage of vinyl ester particulate composites has been reduced by curing the resins under microwave conditions. The reduction in the shrinkage of the resins by microwaves will enable the manufacture of large vinyl ester composite items possible [1-4]. The difference in impact strength between microwave cured vinyl ester particulate composites and those cured under ambient conditions had been investigated and found to be minimal [5]. In addition, a previous study found that the difference in fracture toughness obtained by short bar method between selected microwave-condition cured vinyl ester particulate composites and those cured under ambient conditions was only 0.5% [6]. This project is to investigate the difference in Young's modulus, ultimate tensile strength and yield strength between microwave cured vinyl ester particulate composites and those cured under ambient conditions. The results show that the difference in the Young's modulus is minimal.

1. Introduction

Composite components made from vinyl ester resins by the Centre of Excellence in Engineered Fibre Composites (CEEFC), University of Southern Queensland (USQ) suffer considerable shrinkage during hardening. This shrinkage is particularly serious if the fiber composite components are large. It can be more than ten percent, which is much higher than claimed by some researchers and resins' manufacturers [7, 8]. The main drawback of this shrinkage in a composite component is to have stresses set up internally. These stresses are usually tensile in the core of the component and compressive on the surface [9]. When these stresses act together with the applied loads during service they may cause premature failure of the composite components. Currently, CEEFC solves the shrinkage problem by breaking a large composite

component into smaller composite parts. These smaller parts are then joined together to form the overall structure. By doing this, the manufacturing lead-time and costs of a composite component is significantly increased. The vinyl ester composite used is 33% by weight of fly ash particulate reinforced vinyl ester resins VE/FLYASH (33%), which is exactly the same type of material used in the previous relevant study [1-4].

This research project is to investigate the difference in Young's modulus, ultimate tensile strength and yield strength between microwave cured vinyl ester particulate composites and those cured under ambient conditions. If the mechanical properties of the composites cured under microwave conditions was up to 95% or more than that cured under ambient conditions, microwave treatment can then be used in curing large vinyl ester particulate composites. By this way, not only the shrinkage problem will be solved but also the production time can be shortened [2, 10]. This will eventually lower the production costs of vinyl ester particulate composites. In this project, Australian Standard 1145 – 1989 is used as the basis for determining the tensile properties of the composites [11].

2. Background

2.1 The samples

The vinyl ester resin used is Hetron 922 PAS in summer and Hetron 922 PAW in winter. The vinyl ester is dissolved in 50% by weight of styrene. In this study, Hetron 922 PAW was used. It is based on the reaction between methacrylic acid and

diglycidylether of bishphenol A. The resin hardener ratio used in the experiment was 98% resin by volume and 2% hardener by volume [12]. The reinforcer was fly ash (ceramic hollow spheres) particulate and they were made 44% by volume or 33 % by weight in the cured vinyl ester composite VE/FLYASH (33%). 33 % by weight of flyash in the composite is considered optimum by the CEEFC because the composite will have a reasonable fluidity for casting combined with a good tensile strength in service.

As the raw materials of the composites are liquid and ceramic hollow spheres, the tensile test specimens were cast to shape. The resin is first mixed with the accelerator, methyl ethyl ketone peroxide (MEKP). After that the fly ash is added to the mixture and they are then mixed to give the uncured composite, which was then poured into the moulds (figure 1) of PVC for curing in ambient or microwaved conditions [3]. The samples were made by casting the uncured composites into the moulds.

2.2. Interaction of Microwaves with VE/FLYASH (33%)

Whether a material will absorb microwave energy and convert it into heat depends on its relative complex permittivity and loss tangent. Vinyl ester resin is produced from modified epoxy resin and methacrylic acid and since epoxy resin absorbs microwave irradiation readily, it is therefore expected that it will also absorb microwaves readily [13-15]. A possible risk in applying microwave energy to the vinyl ester composite is the interaction of highly flammable styrene vapour coming out of the resin with the probable arc or heat of the high voltage (HV) transformer in the oven, and can lead to ignition or explosions. To avoid this, the oven (originally Menumaster DEC1800VP)

was modified to ensure that ignition or explosion would not happen and the details of the modifications have been mentioned in another paper [3]. The maximum power output of the oven is 1800 W and power can be step up in 10 steps of 180W. The frequency used is 2.45 GHz.

2.3 Sample Size and Tensile Tests

A MTS 810 Material Testing Systems was used for the test. The capacity of the testing machine is 100 kN and its operating system is Test Star IIs. The rate of extension was 1 mm per minute. In this project, VE/FLYASH (33%) was exposed to microwave irradiation of 180, 360 and 540 W. The duration of exposure for three power levels was 20, 30, 40, 50 and 60 seconds respectively. After pouring the composite into the mould, the mould was located inside the microwave oven cavity, for exposure to microwave irradiation, e.g. exposing the samples in the mould to 40 seconds of microwaves with a power level of 180 W. With the above varying parameters of power levels and exposure of duration in mind, sample size for each set of parameters was made five. At the same time, five similar composite samples were cured under ambient conditions and force required to break them will be used a benchmark for comparison. Tensile tests are performed for various reasons. The results of tensile tests are used in selecting materials for engineering applications. Tensile properties are measured for development of new materials and processes. Tensile properties often are used to predict the behavior of materials under loads other than uniaxial tension [16].

3. Results

3.1 Stress and strain curve

In the tensile test, the force and extension of the test pieces were recorded. Figure 2 shows a typical curve for the specimen undergoing the test. This graph gives the information of tensile force versus tensile elongation.

Engineering stress can be calculated as follows [17]:

$$\sigma = \frac{F}{A_o} \tag{1}$$

where F is the tensile force in Newton and A_o is the initial cross-sectional area of the gauge section in mm^2.

Engineering strain 'ε' can be calculated as follows [17]:

$$\varepsilon = \frac{\Delta L}{L_o} \tag{2}$$

where L_o is the initial gauge length and ΔL is the change in gauge length, $L-L_o$.

3.2 Yield Strength

It is the strength at which a definite amount of plastic strain has occurred As the force-extension curve of the material does not posses a perfectly linear portion (figure 2), the Young's modulus quoted is the secant modulus at a strain of 0.2 percent [9, 18]. Figure 2 also illustrates the results of the test of that particular specimen (40 seconds of exposure duration to microwaves of 180 W), e.g. the peak load is 885 N and the elongation at break is 0.498 mm. Table 1 summarizes the test results of the five samples cured under microwave conditions of 180 W and 40 seconds.

Figure 2 also illustrates how a 0.2% offset line was drawn parallel to the most approximated linear portion of the curve and the intersection of the offset line with the curve. When the intersection was projected to the y-axis, the load found was 885 N which is the 0.2 % offset yield load. Yield strength is calculated using the relationship below [17]:

$$\text{Yield strength} = \frac{Yield\ \ load}{Original\ \ cross-sectional\ \ area} \tag{3}$$

For example, the yield strength of sample 1 exposed to microwave irradiation of 180 W and 40 seconds = $\frac{0.2\%\ \ offset\ \ load}{Original\ \ cross-sectional\ \ area} = \frac{885}{14.8x3.8} = 15.73$ (MPa)

3.3 Tensile strength

This tensile strength can be calculated by dividing the maximum load with the original cross sectional area of the specimen as follows [17]:

$$\text{Tensile strength} = \frac{Maximum\ \ load}{Original\ \ cross-sectional\ \ area} \tag{4}$$

or

$$\sigma = \frac{P_{max}}{A_o} \tag{5}$$

where P_{max} is the maximum load in Newton and A_o is the original cross-sectional area in mm^2.

For example, the tensile strength of sample 1 exposed to microwave irradiation of 180 W and 40 seconds = $\frac{905}{14.8x3.80} = 16.09$ (MPa).

The tensile strength is most sought after result of a tensile test. It is easy to determine and has become a familiar property and is useful for the purposes of specifications and quality control of a product.

3.4 Young's modulus

The Young's modulus (E) or modulus of elasticity is to measure the stiffness of the material. The Young's modulus can be calculated by calculating the slope of the initial linear portion of the stress-strain curve. The Young's modulus [17]:

$$E=\frac{stress}{strain}=\frac{\sigma}{\varepsilon} \tag{6}$$

From (1) and (2), (6) becomes $$E = \frac{\frac{F}{A_o}}{\frac{\Delta L}{L_o}} \tag{7}$$

For example, the Young's modulus of sample 1 exposed to microwave irradiation of 180 W and 40 seconds was calculated using the data provided from figure 3, in which a portion of the most linear part of the curve was selected; after projecting the top point of the selected linear portion into the x- and y- axis respectively, the force (= 453 N) and the extension (= 0.187 mm) were obtained and used in the calculation.

$$E = \frac{\frac{453-0}{14.8x3.8}}{\frac{0.187}{60}} = 2584.41 \text{ (MPa)} = 2.584 \text{ (GPa)}.$$

3.5 Discussions

Table 2 shows yield strength of samples cured under ambient and microwave conditions. Figure 4 illustrates the yield strengths for VE/FLY-ASH (33%) when exposed to three different power levels (180 W, 360 W & 540 W) of microwaves. It can be found that whether it is at 180 W, 360 W or 540 W microwave powers, the yield strengths obtained for all duration of exposure to microwaves are within 5 percent markers that obtained by ambient curing.

Referring to the graph (figure 4) of the yield strengths of test pieces obtained by exposing them to 180W of microwaves, it is found that when duration of exposure is 30 to 50 seconds, the yield strength is higher than that obtained when the composite is cured under ambient conditions. The shrinkage of the composite is least when it is exposed to 180 W microwaves for 35 seconds and it can be observed that at 35 seconds of exposure, the yield strength is approximately the same as that cured under ambient conditions [3, 4]. With these parameters of microwave processing, the total energy dissipated to break the samples in drop weight impact test is also at par with that required to break a sample cured under ambient conditions [5]. These data give a combined outcome of reducing the shrinkage of vinyl ester composite and at the same time maintaining its impact strength and yield strength.

Referring again to the graph (figure 4) of the yield strengths of test pieces obtained by exposing them to 360W of microwaves, it is found that the yield strength is nearly 5 percent higher than that obtained when the composite is cured under ambient conditions. This is encouraging. However, with these parameters of microwave

processing, the total energy dissipated to break the samples in drop weight impact test is 5 percent less than that required to break a sample cured under ambient conditions [5]. This is because some blowholes appeared in the samples and the shrinkage is also larger [3, 4]. Therefore, the parameters used in this microwave processing will enhance the yield strengths of the composites by five percent than that cured under ambient conditions but reduce the impact strength by more than 5 percent.

Referring again to the graph (figure 4) of the yield strengths of test pieces obtained by exposing them to 540W of microwaves, the yield strengths of the test pieces are generally lower than those cured under ambient conditions. Moreover, experiments also proved that the shrinkage of the composite treated by these microwave parameters is more than 5 percent and the impact strength is weak. Ku et al. [3] showed that there were large blowholes in the samples cured under these microwave parameters, which are therefore not recommended for curing vinyl ester composites. Therefore, it can be argued that treating vinyl ester composites with 180 W of microwave irradiation is more beneficial.

In addition, it is worth noting that the values of yield strengths obtained from the experiments of curing the samples with power levels of 180 W, 360 W and 540 W are reliable as the standard deviation of the data is very small and this is shown in table 1.

Table 3 shows tensile strength of samples cured under ambient and microwave conditions. Figure 5 shows the tensile strength for VE/FLY-ASH (33%) when exposed to three different power levels (180 W, 360 W & 540 W) of microwaves. It

can be found that with180 W microwave power, the tensile strengths obtained for all duration of exposure to microwaves are within the 5 percent markers of that obtained by ambient curing. While, with 360 W microwave power, the tensile strengths obtained for all duration of exposure to microwaves are well above the 5 percent markers of that obtained by ambient curing. Whereas, with 540 W microwave power, the tensile strengths obtained for most samples are below the 5 percent markers of those obtained by ambient curing. On top of it, the shrinkage of the composites would also be large, while the impact strength would be lower than the ambient cured counterpart. The 540-W of microwave processing is unfavorable.

Referring to the graph (figure 5) of the tensile strengths of test pieces obtained by exposing them to 180W of microwaves, it is found that with exposure duration of 30 to 40 seconds, the tensile strengths of the test pieces of are almost the same as that cured under ambient conditions. Even though, the tensile strength values of the microwave-cured samples do not higher than its ambient cured counterpart, the microwave parameters used are favourable because they bring about all the positive outcomes as mentioned in the section of yield strength (figure 4). As for the 360 W microwave processing, the tensile strengths and yield strengths (figure 4) obtained are promising but the impact strength is reduced by more than 5 percent. The parameters of microwave treatment of vinyl ester composites in the previous paragraph are therefore better.

Table 4 illustrates the Young's modulus of samples cured under ambient and microwave conditions. Figure 6 shows the Young's modulus of VE/FLY-ASH (33%) when exposed to two different power levels (180 W and 360 W) of microwaves. It is

found that the Young's modulus of the graphs for the three microwave power levels used is 5 percent lower than that cured under ambient conditions. Those exposed to 540 W have their Young's modulus 10 percent lower than that cured under ambient conditions. The Young's modulus for the samples treated with the above microwaves irradiation parameters is reliable because their standard deviation is very small as depicted in Table 4. However, previous work showed that the shrinkage of the composites cured by microwave power of 360 W and 540 W was relatively large, while the impact strength would also be lower than the ambient cured counterpart; the two power levels are therefore not recommended for use [3].

Another interesting point to observe is that when samples of the composites were exposed to microwave power for 50 seconds at a power level of 180 W, their yield and tensile strengths were much higher than those cured under ambient conditions. (figures 4 and 5 respectively). However, their Young's modulus values were slightly below that cured under ambient conditions. It appears to the authors that if lower power levels of microwaves were used, say 100 W to cure the composites with a longer exposure time, it is possible to obtain samples with higher yield and tensile strengths with reduced and acceptable percentage of shrinkage.

4. Conclusions

After studying figures 4 through 6 and my previous paper about the shrinkage reduction of the composite when exposed to microwave irradiation [3], it can be argued that only low power of microwaves, in this case 180W and medium duration of exposure, in this case 30 to 40 seconds, can be used to treat the composite,

VE/FLY-ASH (33%). This treatment will result in smaller shrinkage of the composite specimens together with the same or higher values of yield strength, tensile strength and Young's modulus than those samples cured under ambient conditions. The results obtained in this research project were only preliminary. The results and problems of this research project can be used as a foundation for further work.

If future study were carried out, it is not necessary to expose the composite to power levels beyond 180 W as the mechanical properties obtained will be inferior to those obtained by curing the specimens in ambient conditions. Another worth doing thing is to acquire microwave facilities with lower power levels, e.g. 50 W to 100 W and treat the composite with lower power level but longer duration of exposure; it is anticipated that some promising results can be acquired.

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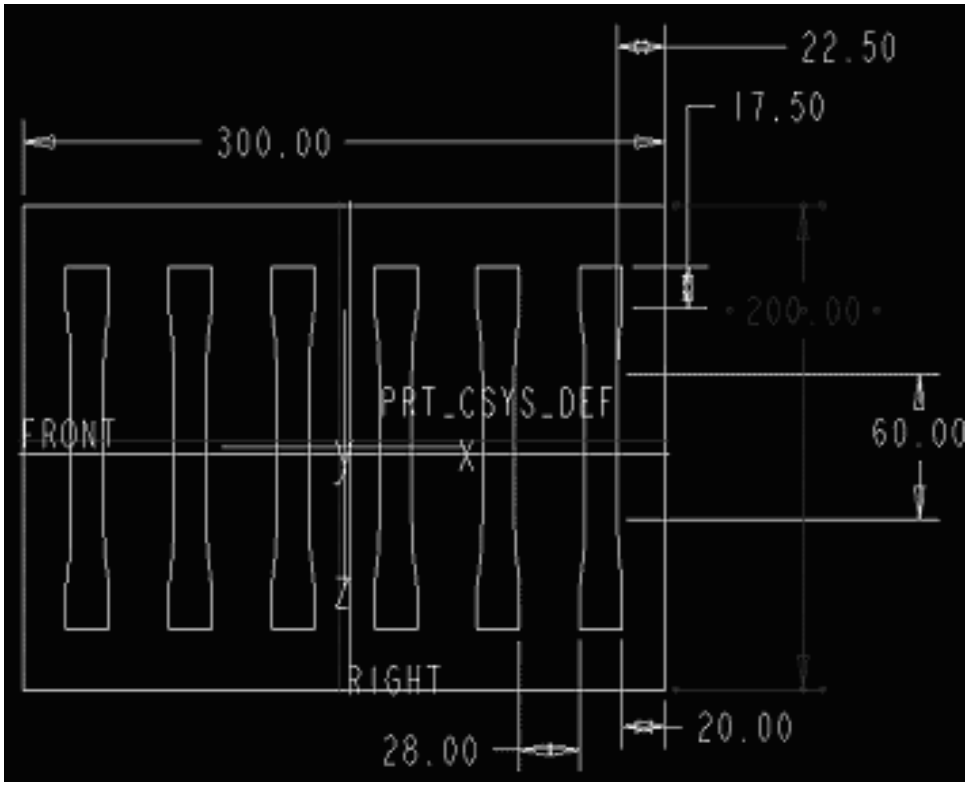

Figure 1: The mould with dimensions

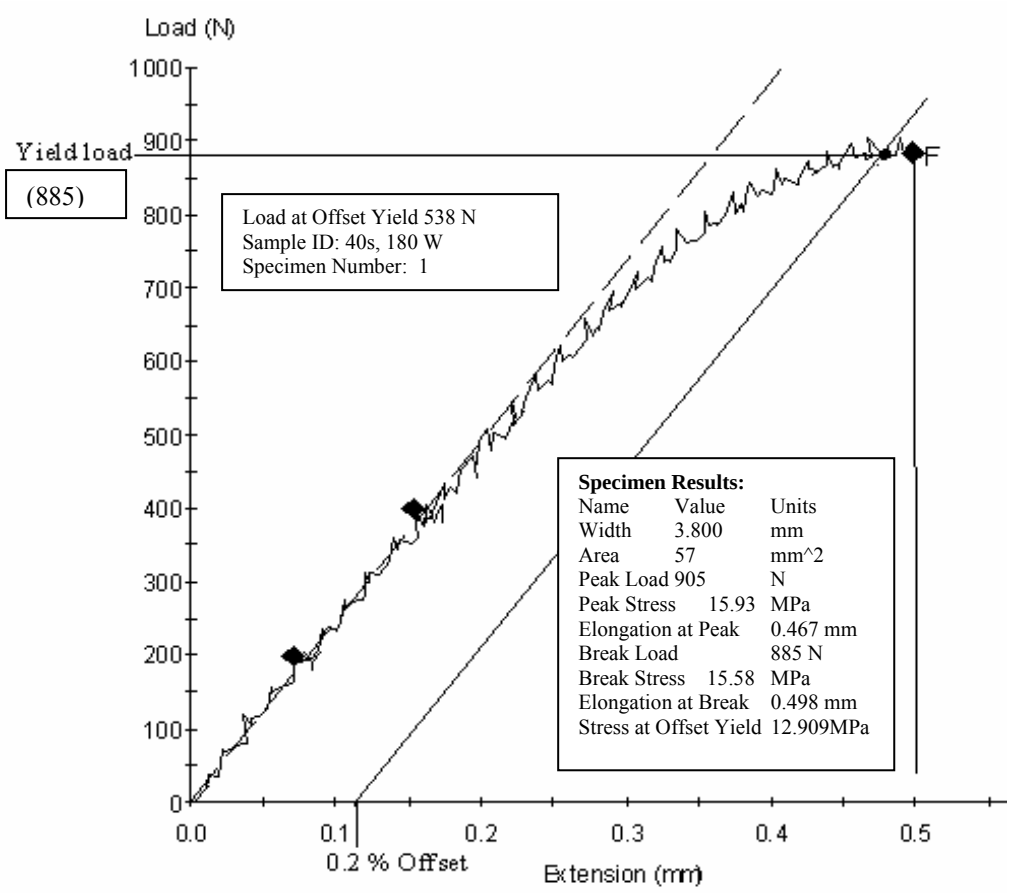

Figure 2: Load against extension of a sample cured under microwave conditions, 180 W, 40 s.

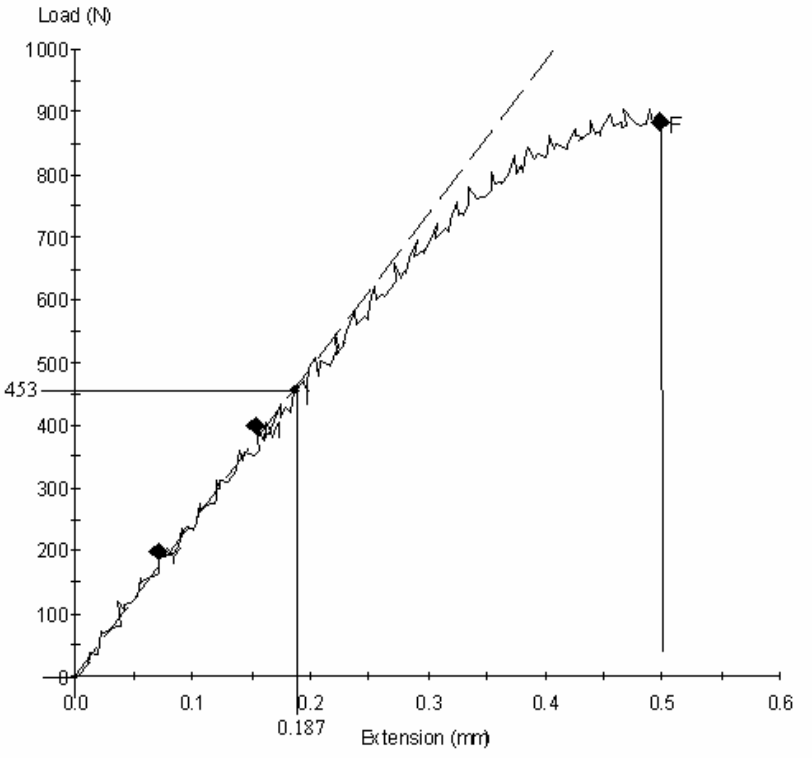

Figure 3: Graph showing how to get data for calculating Young's modulus

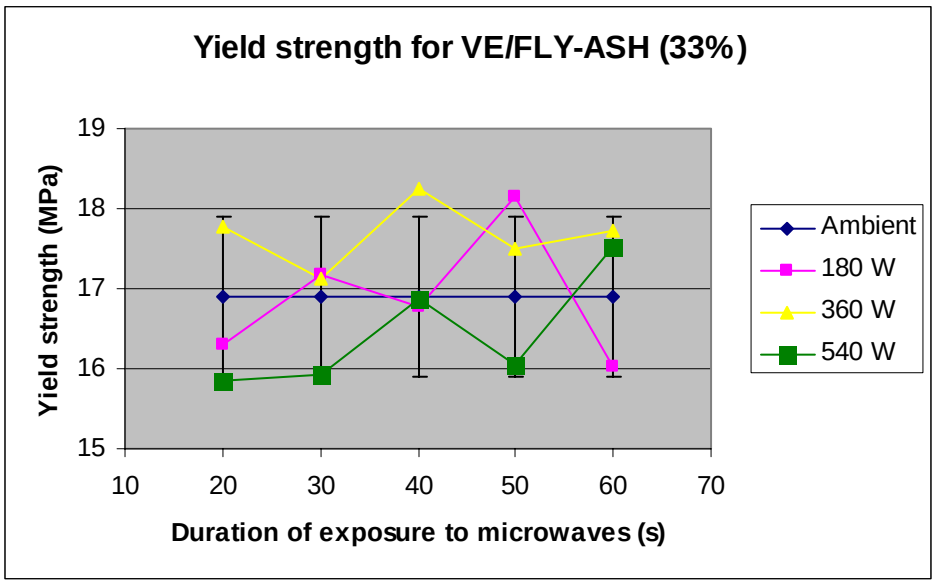

Figure 4: Yield Strength of VE/FLY-ASH (33%) when exposed to three different power levels (180 W, 360 W & 540 W) of microwaves

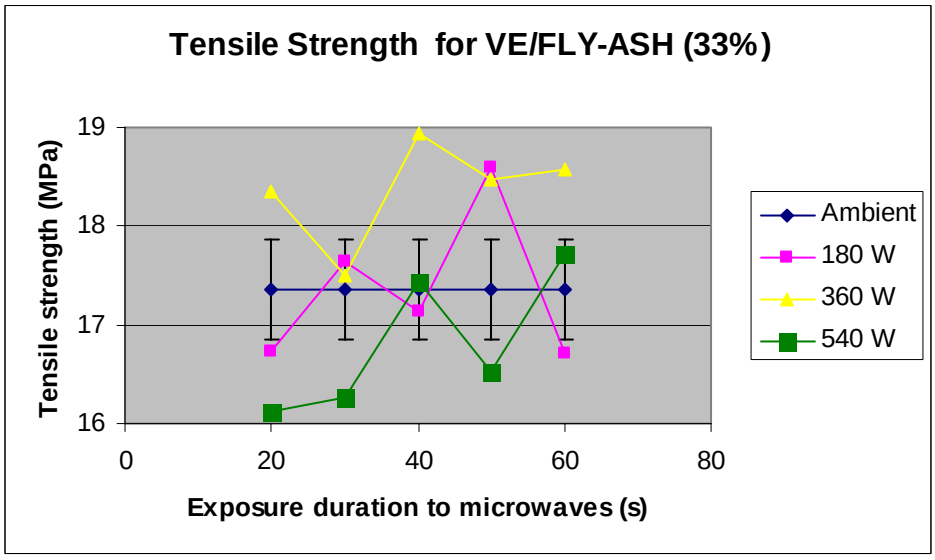

Figure 5: Tensile Strength of VE/FLY-ASH (33%) when exposed to two different power levels (180 W, 360 W & 540 W) of microwaves

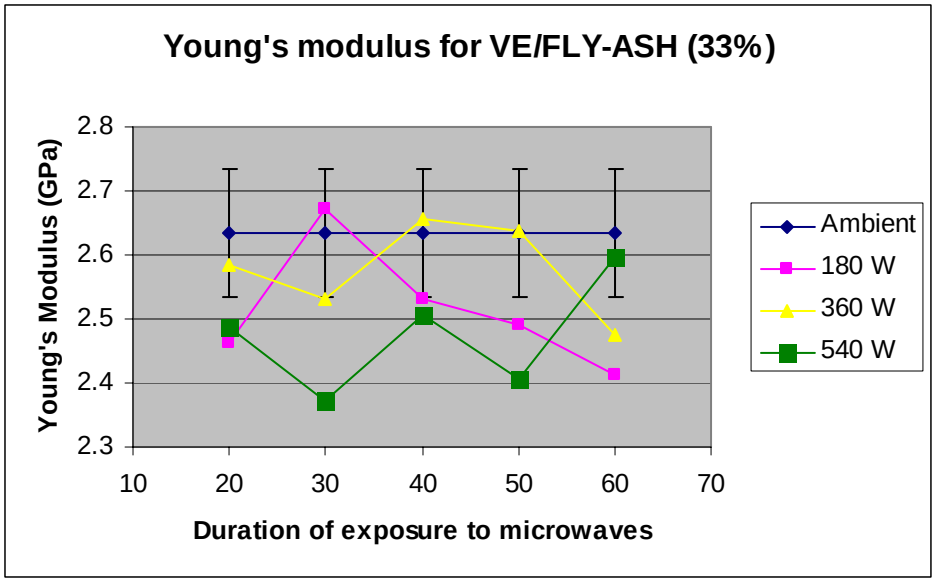

Figure 6: Young's Modulus of VE/FLY-ASH (33%) when exposed to two different power levels (180 W, 360 W & 540 W) of microwaves

Table 1: Test results of the five samples cured exposed to microwave for 40 seconds at 180 W.

Specimen	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Elongation at Peak mm	Break Load N	Break Stress MPa	Elongation at Break mm	Stress at Offset Yield MPa	Load at Offset Yield N
1	14.800	3.800	56	905	16.09	0.467	885	15.74	0.498	13.040	733.368
2	14.800	3.750	55	973	17.54	0.609	945	17.02	0.650	13.350	740.925
3	14.800	3.750	55	943	16.99	0.540	932	16.79	0.562	12.211	677.704
4	14.800	3.850	57	1007	17.68	0.508	959	16.83	0.534	13.432	765.371
5	14.800	3.750	55	962	17.34	0.541	900	16.22	0.655	12.903	716.142
Mean	**14.800**	**3.780**	**56**	**958**	**17.13**	**0.533**	**924**	**16.52**	**0.580**	**12.987**	**726.702**
Std dev	**0.000**	**0.045**	**1**	**38**	**0.63**	**0.052**	**31**	**0.53**	**0.070**	**0.485**	**32.613**

Table 2: Yield strength of samples cured under ambient and microwave conditions

Power levels / Time (s)	Ambient conditions (MPa)	180 W (MPa)	360 W (MPa)	540 W (MPa)
20		16.29(0.21)	15.86(0.84)	16.61(0.38)
30		17.18(0.53)	15.92(2.02)	17.16(0.32)
40	16.9(0.45)	16.77(0.41)	16.87(0.46)	16.9(0.28)
50		18.16(0.8)	16.05(0.06)	16.35(0.69)
60		16.03(0.48)	17.52(0.61)	16.12(0.9)

Table 3: Ultimate tensile strength of samples cured under ambient and microwave conditions

Power levels / Time (s)	Ambient conditions (MPa)	180 W (MPa)	360 W (MPa)	540 W (MPa)
20		16.72(0.34)	18.35(0.48)	16.12(1.62)
30		17.64(0.56)	17.51(1.30)	16.27(3.35)
40	17.36(0.65)	17.13(0.63)	18.94(0.4)	17.43(0.60)
50		18.59(1.06)	18.48(0.6)	16.52(0.11)
60		16.7(0.53)	18.58(0.19)	17.72(1.13)

Table 4: Young's modulus of samples cured under ambient and microwave conditions

Power levels / Time (s)	Ambient condition (GPa)	180 W (GPa)	360 W (GPa)	540 W (GPa)
20		2.464(0.054)	2.583(0.012)	2.488(0.025)
30		2.672(0.027)	2.532(0.1)	2.371(0.12)
40	2.633(0.064)	2.532(0.055)	2.656(0.035)	2.506(0.054)
50		2.492(0.076)	2.636(0.039)	2.407(0.089)
60		2.413(0.037)	2.474(0.035)	2.596(0.114)